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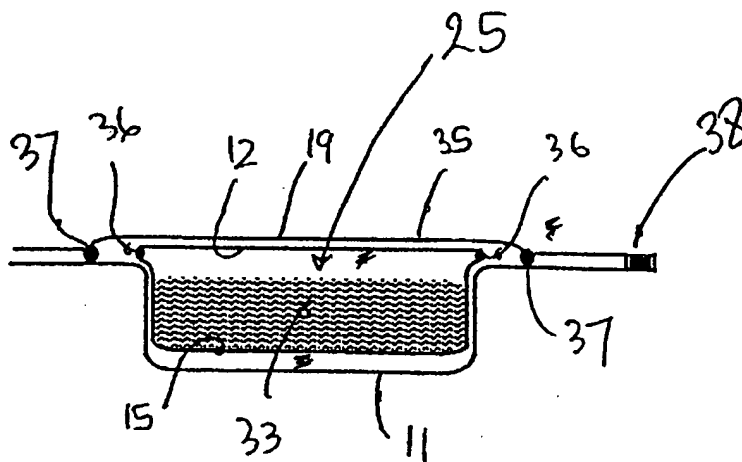
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[Continued on next page]

(54) Title: IMPROVEMENTS IN OR RELATING TO COMPOSITIONS



(57) Abstract: Thermoformed water-soluble packages are known, but some suffer from "shrink-back", which prevents the volume of a formed cavity being fully utilised at the filling stage. To overcome this the present invention proposes a packaging system wherein each individual container (25) of a multiplicity of such containers all joined together in a group side by side in a web comprises an inner water-soluble or dispersible encapsulate (25) enclosed wholly within and thermoformed simultaneously with a protective external water-insoluble casing (19, 11), and each of the encapsulate and the casing is, separately, in the form of a thermoformed hollow body and a lid (23) and is made from two films (15, 12 and 19, 11) bound together by one common seal, so that each encapsulate is in contact only with the inner wall of its casing.

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## IMPROVEMENTS IN OR RELATING TO COMPOSITIONS

This invention is concerned with containers and packaging systems, and relates in particular to a  
5 packaging system incorporating a water-soluble vessel.  
The containers of the invention are particularly useful for pharmaceuticals, pesticides, biocides, compositions which are potentially toxic or damaging or detrimental to health or the environment, and  
10 washing or cleaning materials. As used herein, the term "water-soluble" also encompasses "water-dispersible".

Thermoformed water-soluble packages are known, but some have limitations, one of which is caused by the  
15 propensity of some thermoformed water-soluble films, particularly those comprising a poly(vinyl alcohol) (PVOH) to undergo "shrink-back", which prevents the volume of a formed cavity being fully utilized at the filling stage. The present invention seeks to provide  
20 improved containers, and packaging systems, especially suited for containing pharmaceutical, pesticidal, biocidal, compositions which are potentially toxic or damaging or detrimental to health or the environment, or washing or cleaning compositions.

25 The present invention provides a web comprising a plurality of containers, each container comprising a composition in an internal thermoformed or vacuum formed water-soluble vessel with a water-soluble lid sealed thereto, each sealed vessel being enclosed

within an external thermoformed or vacuum formed water-resistant or water-insoluble holder.

The present invention additionally provides a container comprising a composition in an internal thermoformed or vacuum formed water-soluble vessel with a water-soluble lid sealed thereto, the sealed vessel being enclosed within an external thermoformed or vacuum formed water-resistant or water-insoluble holder.

The present invention also provides a method for making a web or container as defined above which comprises:

- a. positioning a first water-soluble film face to face with a water-resistant or water-insoluble film, the films being in intimate contact so that there is no, or substantially no air trapped between them;
- b. feeding the films on to the mould of a forming machine with the water-resistant or water-insoluble film being closest to the mould;
- c. forming the films in the mould to make an internal vessel cavity and an external holder;
- d. filling the cavity with a composition;
- e. positioning a water-soluble lid over the filled vessel; and
- f. sealing the lid to the water-soluble film.

The method may also optionally comprise:

- g. cutting the water-soluble film and lid to separate at least one of the vessels;
- h. removing an area of waste film produced by the cutting; and/or
- i. sealing a water-insoluble or water-resistant lid to the top of the holder.

5 The present invention furthermore provides the use of a thermoformed or vacuum formed water-resistant or water-insoluble holder to enclose a thermoformed or vacuum formed water-soluble vessel enclosing a composition.

10 More specifically, the present invention provides one or more of an easy-to-make, easy-to-fill and easy-to-use package. For this purpose it proposes a packaging system wherein each individual container of a multiplicity of such containers all joined together in a group side by side in a web comprises an inner water-soluble or -dispersible encapsulate enclosed wholly within a protective external water-insoluble casing, and each of the encapsulate and the casing is, separately, in the form of a thermoformed hollow body and a lid and is made from two films bound together by one common seal, so that each encapsulate is in contact only with the inner wall of its casing. It is possible for the vessel and holder to be in intimate contact over substantially all of the inner surface of the holder. When taking the vessel out of the holder, the user may have a sensation of peeling away the holder from the vessel.

25 In one aspect, therefore, this invention provides a packaging system, useful for making a multiplicity of individual containers all joined together in a group side by side in a web, or an individual container, wherein the container, or each individual container, comprises an inner water-soluble or water-dispersible encapsulate, in the form of a thermoformed vessel and a lid, for example made from two films, especially two

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films bound together by one common seal, enclosed within a protective external water resistant/  
/water-insoluble casing in the form of a thermoformed holder, optionally with a lid, especially a removable  
5 lid such as a peelable lid, and also made from two films bound together by one common seal, so that each encapsulate is in contact only with the inner wall of the casing.

10 In such a packaging system each encapsulate - the inner water-soluble or water-dispersible container, e.g. an envelope - may be filled with any one or more of a wide variety of materials, typical ones being ingredients or compositions useful for  
15 pharmaceuticals, pesticides, biocides, compositions which are potentially toxic or damaging or detrimental to health or the environment, detergents, materials for fabric washing or fabric care, for surface washing or surface care, and for dishwashing, and deodorants, dyes, pigments, or water-treatment chemicals. The  
20 fillings may take any appropriate form - they may for instance be liquids, gels, pastes, solids, granules or powders. The invention's packaging system is, however, most useful for containing liquid compositions. A typical such product is a thickened  
25 liquid detergent formulation.

The material for the walls of the water-soluble/-dispersible compartment - the encapsulate (or envelope) - can be hot- or cold-water soluble or dispersible, and can be flexible or rigid. Preferably,  
30 it is a film, or a combination of two different films, each of which may be a mono-layer or a laminated film,

which is both water-soluble and flexible. By cold-water soluble is meant, for example, a material which is soluble in water at 20°C or less, while by hot-water soluble is meant a material which is soluble in water at 60°C or more. Material which is soluble between these temperatures can also be used. The encapsulate can be made from films of different grades, from films of different thicknesses, or from films which have been perfumed or coloured to obtain the desired characteristics, or from any combination of these.

Preferred materials for the encapsulate are PVOH (polyvinyl alcohol) and cellulose derivatives such as cellulose ethers, for example hydroxypropyl methyl cellulose (HPMC). An example of a preferred PVOH is ethoxylated PVOH. The PVOH may be partially or fully alcoholised or hydrolysed. For example it may be from 40 to 100%, preferably from 70 to 92%, more preferably about 88% or about 92%, alcoholised or hydrolysed. The degree of hydrolysis is known to influence the temperature at which the PVOH starts to dissolve in water. 88% hydrolysis corresponds to a film soluble in cold (ie room temperature) water; whereas 92% hydrolysis corresponds to a film soluble in warm water. They are also generally injection-mouldable, and do not hold a static charge. However, other water-soluble compounds that can be used include polyglycolides and polylactides, and polylactide-polyglycolide copolymers. These materials may of course also, if necessary, contain components such as plasticizers and mould release agents which beneficially modify their

properties - and all of them can naturally include other components such as colouring agents.

5 It has surprisingly been found to be possible by means of this invention to thermoform films which were previously thought to be difficult or impossible to thermoform due to their brittle nature. An example of such a film is an HPMC film.

10 The encapsulate film may be a single film, or a laminated film as disclosed in GB-A-2,244,258. While a single film may have pinholes, the two or more layers of a laminated film are unlikely to have pinholes which coincide.

15 The film may be produced by any process, for example by extrusion and blowing or by casting. The film may be unoriented, monoaxially oriented or biaxially oriented. If the layers in the film are oriented, they usually  
20 have the same orientation, although their planes of orientation may be different if desired.

The layers in a laminate may be the same or different. Thus they may each comprise the same polymer or a  
25 different polymer. If it is desired to have a water-soluble laminated film, each of the layers should be water-soluble.

The thickness of the walls of the encapsulate is  
30 conveniently in the range 20 to 500  $\mu\text{m}$ , preferably 30 to 300  $\mu\text{m}$ , more preferably 35 to 200  $\mu\text{m}$ , especially 40



to 160  $\mu\text{m}$ , more especially 40 to 150  $\mu\text{m}$  and most especially 40 to 120  $\mu\text{m}$ .

5 A typical water-soluble film for the encapsulate's vessel is made of that variety of 75  $\mu\text{m}$  PVOH available as MONOSOL M8534. A typical film for the vessel's lid is made of that variety of 60  $\mu\text{m}$  PVOH available as SOLUBLON PT60.

10 The thermoformed water-insoluble/resistant material for the external protective casing is preferably semi-rigid. Polyester and nylon/polyethylene laminates are convenient for this purpose, preferably in their amorphous form.

15 The thickness of the walls of the water-resistant or insoluble compartment is conveniently in the range of 60 to 1000  $\mu\text{m}$ , preferably 170 to 750  $\mu\text{m}$ .

A typical water-insoluble/resistant film is 170  $\mu\text{m}$  thick and made from amorphous polyester APET; this is suitable for both the holder and its lid.

20 The encapsulate's vessel and the casing's holder are essentially cavities or compartments the one for containing the substance being packaged and the other for containing, and protecting, the first one. These can be of any suitable shape, but preferably they are  
25 each in a slightly tapered cylindrical shape (perhaps with a squarish cross-section), so that a good release from the mould is achieved. In addition, with such a shape one then fits neatly inside the other, so that (as described hereinafter) a set of lined casings

without lid portions can be nested to form a space-saving stack.

In a second aspect the invention provides a method for making containers of the invention, and in particular for making a web or group of such containers joined side by side, in which method:

a. a thermoformable water-soluble or -dispersible lining film, for example in sheet form, is positioned face to face with a thermoformable water-insoluble or -dispersible carrier film in matching sheet form to make a base web combination, the films being in intimate contact such that there is no, or substantially no air trapped between them, and this base web is fed into a thermoformer and there moulded into a sequence of cavity or bowl container shapes with the water-soluble or -dispersible film as a lining on the inside, for each shape the combination being the encapsulate's vessel within the casing's holder;

b. the encapsulate's vessel is filled with the relevant material to be packaged;

c. a water-soluble or -dispersible lid film, for example in sheet form, is placed over the thus-formed lined and filled container shapes, and sealed to the lining film around the mouth of each container shape, forming a lid for the vessel therein, this lidded vessel being the encapsulate, and the lid and lining films are then cut through to separate each encapsulate from its neighbours;

d. the areas of waste lid and lining film between the encapsulates are removed, exposing the underlying carrier film; and, optionally, e. a water-insoluble lid film in matching sheet form is placed over the thus-separated and -spaced casing-borne encapsulates so as to cover them, and is sealed, preferably removably sealed, to the previously-exposed carrier film so as to form for each casing's holder a lid which may preferably be peeled back to expose the encapsulate therein for subsequent removal and use.

The web may be formed on a continuous basis, with a line of, or many side-by-side rows/sets of, containers. How many depends on the width of the sheet; if narrow, like a tape, there might only be one container (so that the method produces a strip of containers), while if wide there might be two, three or more containers in each row. Clearly, the length of the web is indefinite. Once formed, of course, the web can be cut into the more conveniently-sized packs - of individual containers, pairs of containers or two-by-two containers, say - required for sale. If two or more containers are left conjoined, the individual containers may have the same or different sizes, and the compositions held in each container may be the same or different. At that time the holder lidding film, which is preferably peelable, is cut or perforated to allow the lid of each pack to be removed without damaging the hermetic seal of neighbouring packs.

We have surprisingly found that when thermoforming both films together the inner water-soluble film does not undergo as much thinning at corners of the mould as when a film is thermoformed without an outer water-soluble film. This is advantageous since it allows thinner films to be used for the inner vessel, allowing for quicker dissolution in water.

Although in this method the shaping, filling and lidding is most likely to be done all at the same time, it is of course possible to break it into quite widely time-separated stages. Thus, the shaping could be followed by the storing of the formed shapes as "pre-forms", optionally having first cut the sequence of containers into manageable and convenient lengths, and at some later time these pre-forms could be filled, and lidded. Moreover, it is a possibility, though not a preferred one, not to apply the final, water-insoluble or -dispersible film that forms the casing's holder's lid but instead to leave the container as just the encapsulate sitting inside its open-topped holder.

In a thermoforming process the film may be drawn down or blown down into a mould. Thus, for example, the film is heated to the thermoforming temperature using a thermoforming heater plate assembly, and then drawn down under vacuum or blown down under pressure into the mould. Plug-assisted thermoforming and pre-stretching the film, for example by blowing the film away from the mould before thermoforming, may, if desired, be used. One skilled in the art can choose an appropriate temperature, pressure or vacuum and dwell time to achieve an appropriate pocket. The amount of

vacuum or pressure and the thermoforming temperature used depend on the thickness and type of the film and on the polymer or mixture of polymers being used.

Thermoforming of PVOH films is known and described in, for example, WO 00/55045.

A suitable forming temperature for PVOH or ethoxylated PVOH is, for example, from 90 to 130°C, especially 90 to 120°C. A suitable forming pressure is, for example, 69 to 138kPa (10 to 20 p.s.i.), especially 83 to 117 kPa (12 to 17 p.s.i.). A suitable forming vacuum is 0 to 4 kPa (0 to 40 mbar), especially 0 to 2 kPa (0 to 20 mbar). A suitable dwell time is, for example, 0.4 to 2.5 seconds, especially 2 to 2.5 seconds.

While desirably conditions are chosen within the above ranges, it is possible to use one or more of these parameters outside the above ranges, although it may be necessary to compensate by changing the values of the other two parameters.

The inner vessel is then filled with the desired composition. The pocket may be completely filled or only partially filled. The composition may be a solid. For example, it may be a particulate or granulated solid, or a tablet. It may also be a liquid, which may be thickened or gelled if desired. The liquid composition may be non-aqueous or aqueous, for example comprising less than or more than 5% total or free water. The composition may have more than one phase. For example it may comprise an aqueous composition and a liquid composition which is immiscible with the

aqueous composition. It may also comprise a liquid composition and a separate solid composition, for example in the form of a ball, pill or speckles.

5 After the pocket has been filled, a vessel lid such as a film is placed on top of the filled pocket and across the sealing portion, and the films are sealed together at the sealing portion. This film will usually be a single-layered film but may be a laminated film to  
10 reduce the possibility of pinholes allowing leakage through the film. The film may be the same or different as the film forming the cavity. Examples of suitable films are those given for the film forming the cavity.

15 Desirably the vessel film has a thickness which is less than that of the film used for forming a cavity because it would not generally be stretched so localised thinning of the sheet will not occur. It is also  
20 desirable to have a thickness which is less than that of the film used to form a vessel cavity to ensure a sufficient heat transfer through the film to soften the base web if heat sealing is used.

25 The thickness of the covering film is generally from 20 to 160  $\mu\text{m}$ , preferably from 40 to 100  $\mu\text{m}$ , such as 40 to 80  $\mu\text{m}$  or 40 to 60  $\mu\text{m}$ .

30 The sealing effected in the method of the invention can be done by any appropriate means - for example, by an adhesive, heat welding or by ultrasonic, laser,

vibrations, spin, radio frequency or solvent welding, or a combination thereof. Heat sealing, laser sealing and solvent welding or combinations thereof are preferred. A suitable adhesive is water or an aqueous solution of PVOH. The inner seal is desirably water-soluble.

If heat sealing is used, a suitable sealing temperature is, for example, 120 to 195°C, for example 140 to 150°C. A suitable sealing pressure is, for example, from 250 to 600 kPa. Examples of sealing pressures are 276 to 552 kPa (40 to 80 p.s.i.), especially 345 to 483 kPa (50 to 70 p.s.i.) or 400 to 800 kPa (4 to 8 bar), especially 500 to 700 kPa (5 to 7 bar) depending on the heat sealing machine used. Suitable sealing dwell times are 0.4 to 2.5 seconds.

One skilled in the art can use an appropriate temperature, pressure and dwell time to achieve a seal of the desired integrity. While desirably conditions are chosen within the above ranges, it is possible to use one or more of these parameters outside the above ranges, although it might be necessary to compensate by changing the values of the other two parameters.

The composition may be any composition which is intended to be released in an aqueous environment. Thus, for example, it may be a pesticidal composition such as a plant protection agent, for instance an insecticide, fungicide, herbicide, acaricide, or nematocide, or a plant growth regulator, a plant nutrient, or a composition which is potentially toxic or damaging or detrimental to health or the

environment. Such compositions are generally packaged in total amounts of from 0.1 g to 7 kg, preferably 1g to 5 kg, when in solid form. When in liquid or gelled form, such compositions are generally packaged in total  
5 amounts of from 1 ml to 10 litres, preferably 5ml to 6 litres, especially from 10ml to 1.5 litres.

The composition may also be a fabric care, surface care or dishwashing composition. Thus, for example, it may  
10 be a dishwashing, water-softening, laundry or detergent composition, or a rinse aid. Such compositions may be suitable for use in a domestic washing machine. The composition may also be a disinfectant, antibacterial or antiseptic composition, or a refill composition for  
15 a trigger-type spray. Such compositions are generally packaged in total amounts of from 5 to 100 g, especially from 15 to 40 g. For example, a dishwashing composition may weigh from 15 to 30 g, a water-softening composition may weigh from 15 to 40 g.

20

The inner vessel may comprise two or more compartments, particularly if the inner lidding film itself comprises a compartment as disclosed in, for example,  
GB-A-2,361,686.

25

If the article is for use in laundry washing, the primary composition may comprise, for example, a detergent, and the secondary composition may comprise a bleach, stain remover, water-softener, enzyme or fabric  
30 conditioner. The article may be adapted to release the compositions at different times during the laundry wash. For example, a bleach or fabric conditioner is



generally released at the end of a wash, and a water-softener is generally released at the start of a wash. An enzyme may be released at the start or the end of a wash.

5

If the article is for use as a fabric conditioner, the primary composition may comprise a fabric conditioner and the secondary component may comprise an enzyme which is released before or after the fabric conditioner in a rinse cycle.

10

If the article is for use in dish washing the primary composition may comprise a detergent and the secondary composition may comprise a water-softener, salt, enzyme, rinse aid, bleach or bleach activator. The article may be adapted to release the compositions at different times during the laundry wash. For example, a rinse aid, bleach or bleach activator is generally released at the end of a wash, and a water-softener, salt or enzyme is generally released at the start of a wash.

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The primary and secondary compositions, if in liquid form, may independently be anhydrous or comprise water, for example at least 5 wt %, preferably at least 10 wt% free or total water based on the weight of the aqueous compositions. Desirably the compositions contain less than 80 wt% water.

30

The composition may contain surface active agents such as an anionic, nonionic, cationic, amphoteric or zwitterionic surface active agents or mixtures thereof.

Examples of anionic surfactants are straight-chained or branched alkyl sulfates and alkyl polyalkoxylated sulfates, also known as alkyl ether sulfates. Such surfactants may be produced by the sulfation of higher C<sub>8</sub>-C<sub>20</sub> fatty alcohols.

Examples of primary alkyl sulfate surfactants are those of formula:



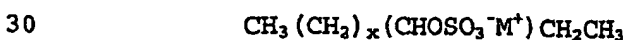
wherein R is a linear C<sub>8</sub>-C<sub>20</sub> hydrocarbyl group and M is a water-solubilising cation. Preferably R is C<sub>10</sub>-C<sub>16</sub> alkyl, for example C<sub>12</sub>-C<sub>14</sub>, and M is alkali metal such as lithium, sodium or potassium.

Examples of secondary alkyl sulfate surfactants are those which have the sulfate moiety on a "backbone" of the molecule, for example those of formula:



wherein m and n are independently 2 or more, the sum of m+n typically being 6 to 20, for example 9 to 15, and M is a water-solubilising cation such as lithium, sodium or potassium.

Especially preferred secondary alkyl sulfates are the (2,3) alkyl sulfate surfactants of formulae:



for the 2-sulfate and 3-sulfate, respectively. In these formulae  $x$  is at least 4, for example 6 to 20, preferably 10 to 16.  $M$  is cation, such as an alkali metal, for example lithium, sodium or potassium.

5

Examples of alkoxyated alkyl sulfates are ethoxylated alkyl sulfates of the formula:



10

wherein  $R$  is a  $C_8$ - $C_{20}$  alkyl group, preferably  $C_{10}$ - $C_{18}$  such as a  $C_{12}$ - $C_{16}$ ,  $n$  is at least 1, for example from 1 to 20, preferably 1 to 15, especially 1 to 6, and  $M$  is a salt-forming cation such as lithium, sodium, potassium, ammonium, alkylammonium or alkanolammonium. These compounds can provide especially desirable fabric cleaning performance benefits when used in combination with alkyl sulfates.

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The alkyl sulfates and alkyl ether sulfates will generally be used in the form of mixtures comprising varying alkyl chain lengths and, if present, varying degrees of alkoxylation.

25

Other anionic surfactants which may be employed are salts of fatty acids, for example  $C_8$ - $C_{18}$  fatty acids, especially the sodium or potassium salts, and alkyl, for example  $C_8$ - $C_{18}$ , benzene sulfonates.

30

Examples of nonionic surfactants are fatty acid alkoxyates, such as fatty acid ethoxyates, especially those of formula:



5 wherein R is a straight or branched C<sub>8</sub>-C<sub>16</sub> alkyl group, preferably a C<sub>9</sub>-C<sub>15</sub>, for example C<sub>10</sub>-C<sub>14</sub>, alkyl group and n is at least 1, for example from 1 to 16, preferably 2 to 12, more preferably 3 to 10.

10 The alkoxyated fatty alcohol nonionic surfactant will frequently have a hydrophilic-lipophilic balance (HLB) which ranges from 3 to 17, more preferably from 6 to 15, most preferably from 10 to 15.

15 Examples of fatty alcohol ethoxylates are those made from alcohols of 12 to 15 carbon atoms and which contain about 7 moles of ethylene oxide. Such materials are commercially marketed under the trademarks Neodol 25-7 and Neodol 23-6.5 by Shell Chemical Company. Other useful Neodols include Neodol 20 1-5, an ethoxylated fatty alcohol averaging 11 carbon atoms in its alkyl chain with about 5 moles of ethylene oxide; Neodol 23-9, an ethoxylated primary C<sub>12</sub>-C<sub>13</sub> alcohol having about 9 moles of ethylene oxide; and Neodol 91-10, an ethoxylated C<sub>9</sub>-C<sub>11</sub> primary alcohol 25 having about 10 moles of ethylene oxide.

Alcohol ethoxylates of this type have also been marketed by Shell Chemical Company under the Dobanol trademark. Dobanol 91-5 is an ethoxylated C<sub>9</sub>-C<sub>11</sub> fatty 30 alcohol with an average of 5 moles ethylene oxide and Dobanol 25-7 is an ethoxylated C<sub>12</sub>-C<sub>15</sub> fatty alcohol with

an average of 7 moles of ethylene oxide per mole of fatty alcohol.

5 Other examples of suitable ethoxylated alcohol nonionic surfactants include Tergitol 15-S-7 and Tergitol 15-S-9, both of which are linear secondary alcohol ethoxylates available from Union Carbide Corporation. Tergitol 15-S-7 is a mixed ethoxylated product of a C<sub>11</sub>-C<sub>15</sub> linear secondary alkanol with 7 moles of ethylene  
10 oxide and Tergitol 15-S-9 is the same but with 9 moles of ethylene oxide.

Other suitable alcohol ethoxylated nonionic surfactants are Neodol 45-11, which is a similar ethylene oxide  
15 condensation product of a fatty alcohol having 14-15 carbon atoms and the number of ethylene oxide groups per mole being about 11. Such products are also available from Shell Chemical Company.

20 Further nonionic surfactants are, for example, C<sub>10</sub>-C<sub>18</sub> alkyl polyglycosides, such as C<sub>12</sub>-C<sub>16</sub> alkyl polyglycosides, especially the polyglucosides. These are especially useful when high foaming compositions are desired. Further surfactants are polyhydroxy fatty  
25 acid amides, such as C<sub>10</sub>-C<sub>18</sub> N-(3-methoxypropyl) glycamides and ethylene oxide-propylene oxide block polymers of the Pluronic type.

30 Examples of cationic surfactants are those of the quaternary ammonium type.

The total content of surfactants in the composition is desirably 60 to 95 wt%, especially 75 to 90 wt%. Desirably an anionic surfactant is present in an amount of 50 to 75 wt%, the nonionic surfactant is present in an amount of 5 to 20 wt%, and/or the cationic surfactant is present in an amount of from 0 to 20 wt%. The amounts are based on the total solids content of the composition, i.e. excluding any solvent which may be present.

The composition, particularly when used as a laundry washing or dishwashing composition, may also independently comprise enzymes, such as protease, lipase, amylase, cellulase and peroxidase enzymes. Such enzymes are commercially available and sold, for example, under the registered trade marks Esperesc, Alcalasc and Savinasc by Nova Industries A/S and Maxatasc by International Biosynthetics, Inc. Desirably the enzymes are independently present in the primary or secondary compositions in an amount of from 0.5 to 3 wt%, especially 1 to 2 wt%.

The composition may, if desired, comprise a thickening agent or gelling agent. Suitable thickeners are polyacrylate polymers such as those sold under the trade mark CARBOPOL, or the trade mark ACUSOL by Rohm and Haas Company. Other suitable thickeners are xanthan gums. The thickener, if present, is generally present in an amount of from 0.2 to 4 wt%, especially 0.5 to 2 wt%.

Compositions used in dishwashing independently usually comprise a detergency builder. Suitable builders are alkali metal or ammonium phosphates, polyphosphates, phosphonates, polyphosphonates, carbonates, bicarbonates, borates, polyhydroxysulfonates, polyacetates, carboxylates such as citrates, and polycarboxylates. The builder is desirably present in an amount of up to 90 wt%, preferably 15 to 90 wt%, more preferable 15 to 75 wt%, relative to the total weight of the composition. Further details of suitable components are given in, for example, EP-A-694,059, EP-A-518,720 and WO 99/06522.

The composition can also optionally comprise one or more additional ingredients. These include conventional detergent composition components such as further surfactants, bleaches, bleach enhancing agents, builders, suds boosters or suds suppressors, anti-tarnish and anti-corrosion agents, organic solvents, co-solvents, phase stabilisers, emulsifying agents, preservatives, soil suspending agents, soil release agents, germicides, pH adjusting agents or buffers, non-builder alkalinity sources, chelating agents, clays such as smectite clays, enzyme stabilizers, anti-limescale agents, colourants, dyes, hydrotropes, dye transfer inhibiting agents, brighteners, and perfumes. If used, such optional ingredients will generally constitute no more than 10 wt%, for example from 1 to 6 wt%, the total weight of the compositions.

30

The builders counteract the effects of calcium, or other ion, water hardness encountered during laundering

or bleaching use of the compositions herein. Examples of such materials are citrate, succinate, malonate, carboxymethyl succinate, carboxylate, polycarboxylate and polyacetyl carboxylate salts, for example with  
5 alkali metal or alkaline earth metal cations, or the corresponding free acids. Specific examples are sodium, potassium and lithium salts of oxydisuccinic acid, mellitic acid, benzene polycarboxylic acids, C<sub>10</sub>-C<sub>22</sub> fatty acids and citric acid. Other examples are  
10 organic phosphonate type sequestering agents such as those sold by Monsanto under the trade mark Dequest and alkylhydroxy phosphonates. Citrate salts and C<sub>12</sub>-C<sub>18</sub> fatty acid soaps are preferred.

15 Other suitable builders are polymers and copolymers known to have builder properties. For example, such materials include appropriate polyacrylic acid, polymaleic acid, and polyacrylic/polymaleic and copolymers and their salts, such as those sold by BASF  
20 under the trade mark Sokalan.

The builders generally constitute from 0 to 3 wt%, more preferably from 0.1 to 1 wt%, by weight of the compositions.

25 Compositions which comprise an enzyme may optionally contain materials which maintain the stability of the enzyme. Such enzyme stabilizers include, for example, polyols such as propylene glycol, boric acid and borax.  
30 Combinations of these enzyme stabilizers may also be employed. If utilized, the enzyme stabilizers



generally constitute from 0.1 to 1 wt% of the compositions.

5 The composition may optionally comprise materials which serve as phase stabilizers and/or co-solvents. Example are C<sub>1</sub>-C<sub>3</sub> alcohols such as methanol, ethanol and propanol. C<sub>1</sub>-C<sub>3</sub> alkanolamines such as mono-, di- and triethanolamines can also be used, by themselves or in combination with the alcohols. The phase stabilizers and/or co-solvents can, for example, constitute 0 to 1 10 wt%, preferably 0.1 to 0.5 wt%, of the composition.

The composition may optionally comprise components which adjust or maintain the pH of the compositions at 15 optimum levels. The pH may be from, for example, 1 to 13, such as 8 to 11 depending on the nature of the composition. For example a dishwashing composition desirably has a pH of 8 to 11, a laundry composition desirable has a pH of 7 to 9, and a water-softening 20 composition desirably has a pH of 7 to 9. Examples of pH adjusting agents are NaOH and citric acid.

In the present invention, if more than one container, is formed at the same time from the same sheet, the 25 containers may then be separated from each other by cutting the portions between them, for example between the sealed portions or flanges. Alternatively, they may be left conjoined and, for example, perforations provided between the individual articles so that they 30 can be easily separated at a later stage, for example by a consumer. If the articles are separated, the flanges may be left in place. However, desirably the

flanges are partially removed in order to provide an even more attractive appearance. Generally the flanges remaining should be as small as possible for aesthetic purposes while bearing in mind that some flange is  
5 required if the articles are in the form of thermoformed containers to ensure the casing film and the lidding film remain adhered to each other. A flange having a width of 1 mm to 8 mm is desirable, preferably 2 mm to 7 mm, most preferably about 5 mm.

10 The containers produced by the process of the present invention, especially when used for a fabric care, surface care or dishwashing composition, may have a maximum dimension of 50 cm, excluding any flanges. For  
15 example, a container may have a length of 1 to 50 cm, especially 3.5 to 4.5 cm, a width of 1.5 to 50 cm, especially 2 to 3 cm, and a height of 1 to 10 cm, especially 1.25 to 1.75 cm.

20 Embodiments of the invention are now described, though by way of illustration only, with reference to the accompanying diagrammatic drawings in which:

Figure 1 shows the stages of the method of the invention;

25 Figure 2 shows that moment in the method during which the waste sealed lid/lining films are removed;

Figure 3 shows a section through a container of the invention; and

Figures 4A&B show a pre-form made during the method of the invention, and a stack of such pre-forms nested together.

For convenience only a few embodiments are described.  
5 However, any one or more of the individual features of these embodiments may have general applicability in all embodiments of the present invention.

Figure 1 shows a water-soluble PVOH film 12 being fed from a roll into the thermoformer 13 (the actual  
10 forming apparatus is not itself shown) in contact with and on top of the carrier film web 11 to form a combined base web 123. The films 12 and 11 pass between rollers (not shown) which place them in intimate contact with substantially no air trapped  
15 between them, before they pass to the thermoformer 13. This combined base web is drawn through the machine without the need for any special unwind or tension control.

In the thermoforming process both the PVOH film 12 and  
20 the carrier film 11 are formed simultaneously. The PVOH film clings to the carrier film, and the carrier film produces a cavity form which holds the formed PVOH film within itself. If the combined base web 123 is examined closely after forming, it will be seen  
25 that the PVOH film is held so well within the carrier film that it is not obvious without close physical examination that there are two different films within the formed base web.

It is important to understand that at this stage of  
30 the process the two films 12,11 can be easily

separated to reveal a well-defined PVOH cavity. If left for a period of time, the formed PVOH film will start to shrink back, but the time required for this to take place is considerably extended, compared to the rate of shrink-back of a PVOH film which has not been "carried" in this way. Full advantage of this is taken when, after forming, the combined base web 123 cavity is then filled (by filling machine 14) with whatever filling (33) is required; the shrink-back of the PVOH film cavity is conditioned by that of the carrier film, which is almost insignificant.

Once filled, there is constructed the desired water-soluble encapsulate by sealing thereover (at the thermoformer's sealing station 16) a second PVOH film (15) as the top web (as noted earlier, the PVOH top web film 15 may be of a different thickness or a different type from the PVOH base web film 12 in order to bestow different properties on the encapsulate). In a typical application, the PVOH top web 15 is thinner than the PVOH bottom web 12 as it has not had to undergo forming (during which some thinning of the film inevitably takes place, particularly at any corners present in the cavity).

The filled and sealed water-soluble encapsulate is still being carried by the carrier film. It is important to understand that, although carried by the carrier film throughout the sealing process, the encapsulate has not become welded to the carrier film during that sealing process. This is because PVOH - or whatever the water-soluble or water-dispersible film material is - is a hydrophilic material, while

the carrier film is (most preferably) a hydrophobic material (most water-insoluble films currently preferred for thermoforming are hydrophobic). As a result, it is not possible to form a heat weld between the carrier film 11 and the PVOH film 12.

Figure 1 shows how the carrier film 11, rather than being put to waste (as in the case of many other carrying devices when their function has been fulfilled), is used as an integral part of the secondary (protective) packaging for the water soluble encapsulate. First, though, it is necessary to separate the individual water-soluble encapsulates making up the sequence of these encapsulates. This is effected by cutting - die-cutting (at cutting station 17) - through the two water-soluble lid and lining films (top and base webs 15,12) around the seals but not through the underlying carrier film 11, and then removing the waste "in-between" material at a rewind station (18) (this is also shown in Figure 2). This die-cutting process is conventional; it is similar to that used in the flat bed die-cutting of self-adhesive labels (in which only the self-adhesive face material is cut, leaving the self-adhesive label adhering to the uncut "siliconed" release material).

Figure 2 shows a plan view of this operation. After cutting the combined water-soluble films (top and bottom webs 15,12) at the die-cutting station 17, leaving the die-cut-around portions (23) in place, the waste (24) is removed upwards to a rewind station 18 leaving behind the now completely-separate water-

soluble encapsulates 25 held in the cavities of the carrier film 11.

Once the water-soluble encapsulates have been die-cut, and the PVOH lid/lining waste removed, a top web (19)  
5 of water-resistant film is sealed over the sequence (at heat-sealing station 100). This top web 19 of water-resistant film, which need not necessarily be of the same material or thickness as the carrier film 11, needs only to be capable of being reliably (and  
10 preferably peelably) sealed to the carrier film for example, by means of an adhesive or welding by heat, ultrasonic, laser, vibration, spin, radio frequency or solvent welding or a combination thereof but during  
15 this process care must be taken not to trap the edges of the flanges of the water-soluble encapsulates in the formed secondary seal. The finished sequence of webbed containers can then be cut into groups, or into individual containers, at a final cutting  
station (111).

20 In Figure 3 there is shown a cross-sectional view of one example of a single container (or pack) according to the invention. The product (33) is enclosed between the two layers of water-soluble lid and lining  
film 15,12 sealed at the periphery (36), and the  
25 resulting water-soluble encapsulate 25 is entirely enclosed within the two water resistant films 11,19, which are sealed around the periphery of the encapsulate with peelable seals (37). This enables  
the outer lid (35) to be readily peeled away from the  
30 carrier film 11, allowing the soluble encapsulate 25 with its product 33 to be elegantly dispensed. If,

for any reason, the lid 35 is required to remain attached to the carrier film 11 after peeling open the pack, the lid may be additionally sealed on one side only with a further seal (38) which is not peelable.

5 Typically, the top web of water resistant film 19 is sealed to the carrier film 11 with a peelable seal which, whilst hermetically sealing the pack from ingress of contaminants, particularly water, also allows a pull tab or other opening device (not shown)  
10 located at the corner of each individual compartment of the pack to be used to gain easy access to the water-soluble encapsulate 25 contained within. The product can then be unit dosed elegantly, leaving the remainder of the encapsulates protected as in a  
15 pharmaceutical blister pack.

A further embodiment of the invention is one where the carrier film 11 is used as the only component of the protective packaging of the water-soluble encapsulate 25. It is still necessary to separate the  
20 water-soluble encapsulates by die-cutting or stamping through the two PVOH films (top and base webs 15,12) around the seals but not through the carrier film and, in the case of die-cutting, the waste PVOH lid/lining films are again removed at a rewind station 18.

25 However, here the process is interrupted after die-cutting such that the second web of water-resistant film 19 is not applied. The carrier film 11 within which the water-soluble encapsulates 25 reside after die-cutting may be retained and cut into trays  
30 containing a plurality of packs for stacking or display purposes.

A yet further embodiment of the invention is described with reference to Figure 4. The process illustrated in Figure 1 is interrupted before the filling stage 14, and the web is moved directly on to the cutting and separation stages 17,18 in order to produce empty so-called "pre-forms" (41) for storage and later use. Figure 4 shows an example of such a pre-form, and how several of these can be nested for efficient storage provided that the cavity has a sufficient taper, narrowing towards the base. It is evident that the pre-forms, complete with their PVOH liner, can be stored, and then at a later date filled with product and fed through a simple lidding and cutting machine (such as those produced by Tiromat and Multivac) to produce the water-soluble encapsulates which are each individually supported by the carrier film but not completely protected by it.

The packaging system and containers of the invention have many advantages, some of them being as follows:-

- a) Shrinkage of the water-soluble/dispersible film between forming and sealing is virtually eliminated.
- b) The water-soluble/dispersible film can be thermoformed without losing its shape and volume due to shrink-back.
- c) The process can be run at increased speeds with reduced risk of contamination of the seals by spillage.



d) The finished pack greatly improves storage, handling and dispensing of the product.

5 e) PVOH films with otherwise desirable properties but especially susceptible to shrink-back can be used efficiently.

10 f) As the PVOH base web film is supported by the carrier film, the suction holes in the cavity forming mould will not be in contact with the PVOH film, thus eliminating any risk of damage to the PVOH film during forming.

15 g) The formed cavities of carrier & PVOH films combined may be separated, singly or in a plurality of units, before filling with product. These unfilled cavities may then be stored and used at a later date. These "pre-forms" will nest and store economically, and require less  
20 sophisticated equipment to fill and seal.

25 h) A package can be produced where the carrier film is the required packaging and the PVOH provides a "barrier" lining. The package will then be able to contain many products that the carrier film alone may not be able to contain, such as perfumes, odours, oils, fats and greases. The package will also act as a barrier to oxygen.

## CLAIMS

1. A web comprising a plurality of containers, each container comprising a composition (33) in an internal thermoformed or vacuum formed water-soluble vessel (25) with a water-soluble lid (12) sealed thereto, each sealed vessel (25) being enclosed within an external thermoformed or vacuum formed water-resistant or water-insoluble holder (11).
2. A web according to claim 1 wherein each holder is provided with a lid (35) sealed thereto.
3. A web according to claim 2 wherein the holder lid (35) is removably sealed to the holder (11).
4. A web according to claim 2 or 3 wherein the holder lid is water-soluble.
5. A web according to any one of the preceding claims wherein the water soluble vessel comprises a poly(vinyl alcohol) (PVOH) or cellulose derivative.
6. A web according to any one of the preceding claims wherein the thickness of the vessel walls is from 20 to 500  $\mu\text{m}$ .
7. A web according to any one of the preceding claims wherein the thickness of the vessel walls is from 40 to 120  $\mu\text{m}$  and the thickness of the vessel lid is from 20 to 80  $\mu\text{m}$ .

8. A web according to any one of the preceding claims wherein the holder is semi-rigid.

5 9. A web according to any one of the preceding claims wherein the holder comprises a polyester or a nylon/polyethylene laminate, in amorphous form.

10 10. A web according to any one of the preceding claims wherein the thickness of the walls of the holder is from 60 to 1000  $\mu\text{m}$ .

11. A web according to claim 10 wherein the thickness of the walls of the holder is from 120 to 750  $\mu\text{m}$ .

15 12. A web according to claim 10 or 11 wherein the thickness of the walls of the holder is about 170  $\mu\text{m}$  and the walls comprise an amorphous polyethylene terephthalate.

20 13. A web according to any one of the preceding claims wherein the vessel and holder are each of a nestable, tapered cylindrical shape.

25 14. A web according to any one of the preceding claims wherein the vessel and holder are in intimate contact over substantially all of the inner surface of the holder.

30 15. A web according to any one of the preceding claims wherein the composition is a liquid composition.

16. A web according to any one of the preceding claims wherein the composition is a fabric care, surface care or dishwashing composition.

5 17. A web according to claim 16 wherein the composition is a dishwashing, water-softening, laundry or detergent composition or a rinse aid.

10 18. A web according to claim 16 wherein the composition is a disinfectant, antibacterial or antiseptic composition, or a refill composition for a trigger-type spray.

15 19. A container comprising a composition in an internal thermoformed or vacuum formed water-soluble vessel with a water-soluble lid sealed thereto, the sealed vessel being enclosed within an external thermoformed or vacuum formed water-resistant or water-insoluble holder.

20 20. A container according to claim 19 which has the features of any one of claims 2 to 18.

25 21. A container according to claim 19 or 20 which contains a unit dose of the composition.

22. A method for making a web or container as defined in any one of the preceding claims which comprises:

30 a. positioning a first water-soluble film face to face with a water-resistant or water-insoluble film

the films being in intimate contact such that there is no, or substantially no air trapped between them;

b. feeding the films onto the mould of a forming machine with the water-resistant or water-insoluble film being closest to the mould;

c. forming the films in the mould to make an internal vessel cavity and an external holder;

d. filling the cavity with a composition;

e. positioning a water-soluble lid over the filled vessel; and

f. sealing the lid to the water-soluble film.

23. A method according to claim 22 which further comprises:

g. cutting the water-soluble film and lid to separate at least one of the vessels.

24. A method according to claim 23 which further comprises:

h. removing an area of waste film produced by the cutting.

25. A method according to any one of claims 22 to 24 which further comprises:

i. sealing a water-insoluble or water-resistant lid to the top of the holder.

26. A method according to any one of claims 22 to 25 which forms a web comprising a plurality of containers

27. A method according to claim 26 wherein the web comprises a two-dimensional array of containers and is formed on a continuous basis.

5 28. A method according to claim 26 or 27 wherein a web is cut into individual containers or smaller groups of containers.

10 29. Use of a thermoformed or vacuum formed water-resistant or water-insoluble holder to enclose a thermoformed or vacuum formed water-soluble vessel enclosing a composition.

Figure 1

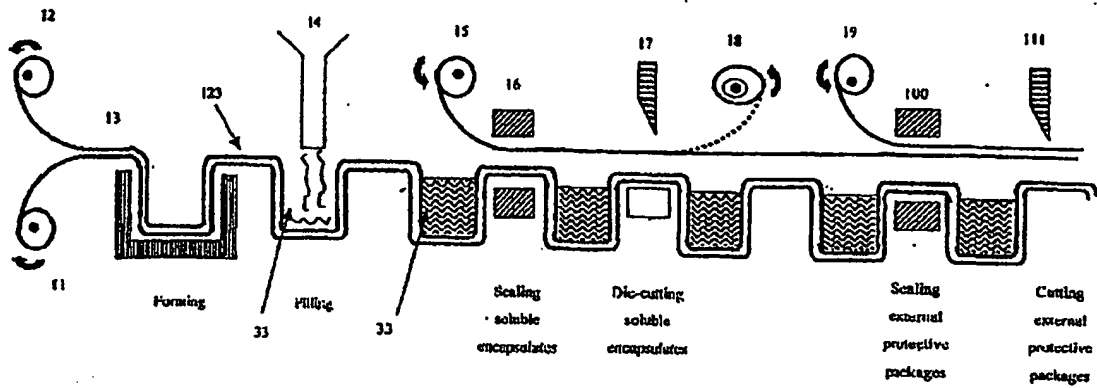


Figure 2

